Department of Pesticide Regulation

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SUBJECT: DETERMINATION IF THE CALIFORNIA DEPARTMENT OF FOOD AND AGRICULTURE, CENTER FOR ANALYTICAL CHEMISTRY'S HIGH PERFORMANCE LIQUID CHROMATOGRAPHY-TANDEM MASS SPECTROMETRY METHOD (EMON-SM-13.0) FOR IMIDACLOPRID, IMIDACLOPRID OLEFIN, IMIDACLOPRID UREA, IMIDACLOPRID GUANIDINE, AND IMIDACLOPRID GUANIDINE OLEFIN MEETS THE "UNEQUIVOCAL DETECTION" CRITERIA

Background

The Pesticide Contamination Prevention Act (Food and Agriculture Code [FAC] sections 13141 et seq.) was passed in 1985 to prevent further pesticide pollution of ground water, which may be used for drinking water supplies. FAC section 13149 specifies the conditions under which a pesticide is considered "found" in ground water or soil, and thus subject to formal review as specified. As originally adopted, FAC subsection 13149(d) specified that a pesticide detection should be verified by a second analytical method or a second analytical laboratory approved by the Department of Pesticide Regulation. However, Senate Bill 810 amended the law in 1995 to allow a finding of a pesticide in ground water or soil based on a single analytical method conducted by a single analytical laboratory, if the analytical method provides unequivocal identification of a chemical. Following this change, general criteria were established to identify methods providing unequivocal identification of a chemical (Biermann, 1996).

Issue

Does the California Department of Food and Agriculture (CDFA), Center for Analytical Chemistry's high performance liquid chromatography-tandem mass spectrometry (HPLC-MS) method EMON-SM-13.0 (Lee, 2008) for imidacloprid and its degradates, meet the definition of an "unequivocal detection" method?

Discussion and Recommendation

CDFA method EMON-SM-13.0 uses a HPLC coupled to an electrospray ionization MS detector. Prior to injection of sample into the HPLC-MS apparatus, the well water samples are cleaned and extracted using solid phase extraction. Consequently the well water samples generally contain a minimal amount of background/matrix interference, facilitating the goal of unequivocal detection.

In CDFA method EMON-SM-13.0 the following criteria are used to confirm the presence of imidacloprid and each of its four degradates:

1. The high performance liquid chromatograph separates each compound according to its characteristic retention time, where the retention time is required to be within twenty seconds of that observed with authentic standard.
2. Each analyte has a precursor/parent ion and corresponding product/daughter ion that needs to be present in order to quantify that analyte.
3. The analyte's ratio of product ion to precursor ion shall be within 20 percent of that observed with authentic standard.
4. The peak response of each of the unknown analytes is required to be within the linear range of the initial calibration curve.

If an analyte observes the above criteria of method EMON-SM-13.0, then that analyte has been unequivocally identified in the sample. Consequently, analysis of imidacloprid and its degradates by this method qualifies for the unequivocal detection designation.

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References

Biermann, H., 1996. Memorandum to K. Goh, "Definition of 'unequivocal detection methods' for the purposes of Senate Bill 810."

Lee, P., 2008. Determination of Imidacloprid and the Olefinic Imidacloprid, Guanidine, Olefinic Quanidine, Urea Metabolites in Well Water by High Performance Liquid Chromatography Tandem Mass Spectrometry. Method EMON-SM-13.0. Available at:<http://www.cdpr.ca.gov/docs/emon/pubs/anl_methds/imeth_308.pdf>.